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IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicants: Rand David Dannenberg
Title: Optical Coatings and Associated Methods
Application No.: 09/944,050 Filing Date: August 30, 2001
Examiner: Chang, Audrey V. Group Art Unit: 2872
Docket No.: VONA.004US0 Conf. No.: 8351

RECEIVED

Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

FEB 24 2004

DECLARATION OF RAND DAVID DANNENBERG

Sir:

I, Rand David Dannenberg, declare under the penalty of perjury as set forth below.

1. I am the inventor named in the above-referenced application ("Application").

During a period prior to July 11, 2000, I was employed by The BOC Group, Inc. ("BOC"), the company to which I assigned invented subject matter that relates to the Application and the Application itself. A copy of the assignment, as recorded by the United States Patent and Trademark Office at Reel 012446 and Frame 0869, is appended hereto as Document A.

2. During my employment at BOC, I conceived of, worked on, and supervised others' work in relation to various inventions, and prepared and internally submitted various invention records. In this capacity, I internally submitted an invention record ("Invention Record") concerning subject matter that relates to the Application, identifying myself as the inventor of that subject matter, and signing and dating the Invention Record, on a date prior to July 11, 2000. The Invention Record was witnessed by a witness, and signed and dated by the witness, on a date prior to July 11, 2000. A copy of the Invention Record, unnecessary

portions of which have been redacted, is appended hereto as Document B. Document B shows the witness' signature in the signature block for witnesses and my signature in the signature block for submitters, although the dates associated with these signatures, each of which reflects a date prior to July 11, 2000, have been redacted.

3. The Invention Record disclosed a problem, namely, that some multi-layer coatings do not survive heat treatment because a layer of the coating crystallizes, and a solution to that problem, namely, preparing a multi-layer coating in which the aforementioned layer is replaced by a layer that comprises an amorphous oxide of titanium and another species, which, in an oxidized state, is insoluble in the oxide of titanium, or forms no solid solution with the oxide of titanium. See Document B, the Invention Record, which references an attachment, and Document C, the attachment, unnecessary portions of which have been redacted. Document C bears a date, which has been redacted, that is prior to July 11, 2000.

4. The Invention Record also included an attachment that documented experiments in which amorphous materials, comprising an oxide of titanium and silicon, were produced via sputtering in environments that comprised oxygen. See Document B, the Invention Record, which references an attachment, and Document D, the attachment, unnecessary portions of which have been redacted. Each page of Document D was signed and dated either by a preparer of the page other than myself, who was under my supervision, and myself, as a witness who had read and understood the material on the page; or by myself, as the preparer of the page, and a witness other than myself who had read and understood the material on the page. Each page of Document D shows the preparer's signature and the witness' signature, in associated signature blocks, although the dates associated with these signatures, each of which reflects a date prior to July 11, 2000, have been redacted.

5. The foregoing subject matter ("Subject Matter of the Invention Record") described above in items 3 and 4, including the foregoing solution and the foregoing experiments, was conceived and reduced to practice before July 11, 2000.

6. The Invention Record, which was submitted, witnessed, signed and dated prior to July 11, 2000, as described above, was kept among usual business records of BOC in the ordinary course of business.

7. Pending claims 1-6 and 16-21, which are currently under consideration in the Application, relate to the Subject Matter of the Invention Record. A copy of these claims is appended hereto as Document E.

8. In view of the foregoing, the invention as reflected in claims 1-6 and 6-21 of the Application was conceived and reduced to practice before July 11, 2000.

9. I further declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishment by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

Executed by myself, Rand David Dannenberg, at the location and on the date set forth below.

Location: Thousand Oaks, California
City

Date: Feb 10, 2004
Month and Day

Signature: Rand David Dannenberg
Rand David Dannenberg

Document A

Assignment Recorded in United States Patent and Trademark Office

F

01-14-2002



101945723

3 SHEET

U.S. DEPARTMENT OF COMMERCE
Patent and Trademark Office

ATTY. DKT. M00A226

To the Honorable Commissioner of Patents and Trademarks: Please record the attached original documents or copy thereof.

1. Name of conveying party(ies):

Rand David DANNENBERG

1.8.02

Additional name(s) of conveying party(ies) attached? ☐ Yes ☒ No

2. Name and address of receiving party(ies):

Name: The BOC Group, Inc.

Internal Address: _____

Street Address: _____

575 Mountain Avenue

Murray Hill

City: New Providence State: NJ ZIP: 07974

Additional name(s) & address(es) attached? ☐ Yes ☒ No

3. Nature of conveyance:

☒ Assignment

☐ Security Agreement

☐ Other _____

☐ Merger

☐ Change of Name

Execution Date: November 26, 2001

4. Application number(s) or patent number(s):

If this document is being filed together with a new application, the execution date of the application is: _____

A. Patent Application No.(s)

09/944,050

filed August 30, 2001

B. Patent No.(s)

Additional numbers attached? ☐ Yes ☒ No

5. Name and address of party to whom correspondence concerning document should be mailed:

Name: Philip H. Von Neida

Internal Address: Intellectual Property Department

Street Address: The BOC Group, Inc.

100 Mountain Avenue

City: Murray Hill State: NJ Zip: 07974

6. Total number of applications and patents involved: 1

7. Total fee (37 CFR 3.41): \$ 40.00

☐ Enclosed

☒ Authorized to be charged to deposit account

8. Deposit account number: 02-2865

(Attach duplicate copy of this page if paying by deposit account)

DO NOT USE THIS SPACE

9. Statement and Signature.

To the best of my knowledge and belief, the foregoing information is true and correct and any attached copy is a true copy of the original document.


Philip H. Von Neida

Registration No. 34,942

December 7, 2001
Date

Total number of pages comprising cover sheet: 1

PATENT
REEL: 012446 FRAME: 0869

SOLE ASSIGNMENT

WHEREAS, I, Rand David Dannenberg, a citizen of the United States of America, residing at 214 East F Street, Benicia, California 94510, USA, have invented certain new and useful improvements in OPTICAL COATINGS AND ASSOCIATED METHODS for which I have made application for Letters Patent of the United States, which application may be identified in the United States Patent Office as Serial No. 09/944,050, filing date August 30, 2001; and,

WHEREAS, The BOC Group, Inc., a Delaware Corporation having an office at 575 Mountain Avenue, Murray Hill, New Providence, New Jersey 07974, is desirous of obtaining the entire right, title and interest in, to and under the said improvements and the said application:

NOW, THEREFORE, in consideration of the sum of One Dollar (\$1.00) to me in hand paid, and other good and valuable consideration, the receipt of which is hereby acknowledged, I, the said Rand David Dannenberg have sold, assigned, transferred and set over, and by these presents do hereby sell, assign, transfer and set over, unto the said The BOC Group, Inc., its successors, legal representatives and assigns, the entire right, title and interest in, to and under the said improvements, and the said application and all divisions, renewals and continuations thereof, and all Letters Patent of the United States which may be granted thereon and all reissues and extensions thereof, and all applications for Letters Patent which may hereafter be filed for said improvements in any country or countries foreign to the United States, and all Letters Patent which may be granted for said improvements in any country or countries foreign to the United States and all extensions, renewals and reissues thereof; and I hereby authorize and request the Commissioner of Patents of the United States, and any Official of any country or countries foreign to the United States, whose duty it is to issue patents on applications as aforesaid, to issue all Letters Patent for said improvements to the said The BOC Group, Inc., its successors, legal representatives and assigns, in accordance with the terms of this instrument.

I HEREBY covenant that I have full right to convey the entire interest herein assigned, and that I have not executed, and will not execute, any agreement in conflict herewith.

AND I HEREBY further covenant and agree that I will communicate to the said The BOC Group, Inc., its successors, legal representatives and assigns, any facts known to me respecting said improvements, and testify in any legal proceeding, sign all lawful papers, execute all divisional, continuing and reissue applications, make all rightful oaths, and generally do everything possible to aid the said The BOC Group, Inc., its successors, legal representatives and assigns, to obtain and enforce proper patent protection for said improvements in all countries.

IN TESTIMONY WHEREOF, I hereunto set my hand and seal this 26 day of NOVEMBER, 2001.

State of California
County of Solano ss.:

Rand David Dannenberg
Rand David Dannenberg

On this 26 day of November, 2001, before me, a Notary Public in the State and County aforesaid, personally appeared Rand David Dannenberg, to me known and known to me to be the person of that name, who signed and sealed the foregoing instrument, and he acknowledged the same to be his free act and deed.



Mary L. Richardson
Notary Public

RECORDED: 01/08/2002

PATENT
REEL: 012446 FRAME: 0870

Document B

Invention Record with Portions Redacted

INVENTION RECORD

An amorphous oxide film of Ti and another metallic species is deposited by an energetic process. The crystallization temperature of the amorphous film and also the index of refraction is controlled by the dopant and doping level. The material is intended to be used in optical coatings which can withstand heat treatment to various temperatures below the crystallization temperature of the compound, such as anti-reflection coatings and low-emissivity coatings.

TiO₂ is "doped" with another metallic species (X=Si, Al, Bi, Gd, Ta, Zn and others) and deposited under conditions in which the film has an amorphous (glassy) structure, called a-TiXO. The second material, in the oxidized state, called XO_x, must be insoluble in TiO₂ (form no solid solutions).

One example is Ti-Si-O. TiO_2 and SiO_2 are insoluble and form no solid solutions.

ASSERTION OF INVENTORSHIP: (Name(s) of person or persons, if any, who claim inventorship.)

Rand Dannenberg.

CONCEPTION DATE:

See attached report

DATE OF ACTUAL REDUCTION TO PRACTICE:

See photocopy

WITNESSES:

Date:

Signature:

Russell S. Hill

Date:

Signature:

Date:

Signature:

SUBMITTED BY:

Date:

Signature:

Ran D. Dancy
Title

Date:

Signature:

Title

Date:

Signature:

Title

Document C

Attachment to Invention Record with Portions Redacted and First and Third Pages Fully Redacted

I have been experimenting with a rate enhanced $\alpha\text{-TiO}_x/\text{Ti}/\text{Ag}/\text{Ti}/\alpha\text{-TiO}_x$ multilayer with poor results after heating to 650 °C. One reason that the coating may not be surviving the heat treatment is because the top layer of $\alpha\text{-TiO}_x$ crystallizes at 300 °C, and in doing so, forms not only grain boundaries which enhance diffusion, but also causes the film to change size and partially delaminate.

solution involves doping the target material with an element in which both compounds would be mutually insoluble in the reacted state (i.e. forms no solid solutions).

Document D

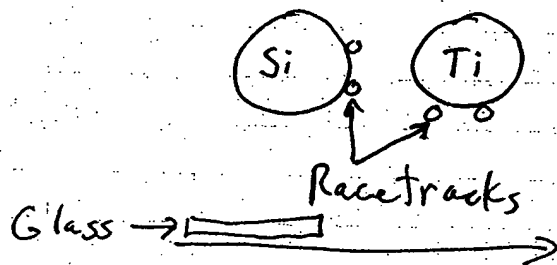
Attachment to Invention Record with Portions Redacted and Second Page Fully Redacted

Si doped Ti, Co Sputtered

○

Si sputtered onto Ti C-Mag in R4, SB2

End view of tubes showing race track locations.



First, start off to see if we can run this set-up with 7Kv on the Ti tube. This is desired to get an amorphous film.

10 sccm Ar purge on PEM in SB3

85 sccm O₂ in SB2 (where the cathode is) → 3.0 mT

It runs at 1 Kv on Ti, 0.3 Kv on S

- Need to boost O₂ flow to 100 sccm for pressure at 3.3 mT

Read and Understood By

[Signature]

Signed

Date

Date

Signed

[Signature]

Si doped Ti, Co ~~O~~ patterned

Go back to the 5KV Ti, 1.0 kW Si run and
run samples on TEM grids (2) and VWR slide (2).

27%
250 Å

1 pass at 7.4 in/min

Sample K.H.10 box 54511

(This was based on 27% Si parameters)

576-96-1

- Too thin for RET analysis in WVR

Gas still 10 sccm Ar purge

40 sccm O₂

25 sccm Ar

Ti @ 5.0 kW

Si @ 0.3 kW

3 passes at 6.2 in/min

576-96-2

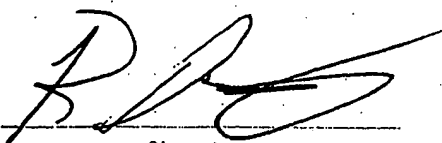
742 Å, DDR = 106, Gradient = -3.7
at 550 nm, $n = 2.314$ $K = 0.0012$
 $\Rightarrow \alpha = 13\% \text{ Si}$

13% 1/4 wave

Make a TEM set based on above @ 13%
6.1 in/min 1 pass, 576.96.3

13% 250 Å


(K.H. 8 ~~box~~ box 54511)



Signed

Date

Read and Understood By



Signed

Date

As per 95-2 100% TiO_2 , but 5.3 μm 250 Å TEH's KMO 6
5 kW gas as per pg. 94 376.94.1

100% TiO_2 250 Å

95.2 0% Si \rightarrow 643 Å $n = 2.433$

97.1 0% Si \approx 250 Å \rightarrow good fit some constants

96.2 13% Si \rightarrow 744 Å $n = 2.315$

96.3 13% \approx 250 Å \rightarrow good fit some constants

95.1 27% Si \rightarrow 906 Å $n = 2.183$ (some absorption)

96.1 27% Si \approx 250 Å (better fit to about 200 Å, but okay)
(206 Å) about a 45 Å predicted difference)

if index n is high, d low n is d (if fix 250 Å $n_{\text{resonance}} = 2.09$)

Read and Understood By

R. J. Tausch
Signed

Date

Peter J. Tausch
Signed

Date

Specimen 96-2/963 $\approx 13\%$ Si box SASII K8
Film is anorphous

begin filming at 9:04

- no sign of nucleation/segregation up to 120°C
- 200°C many small particles beginning to appear!
- does not seem to be continuing.
- 90 to 300°C
- nothing interesting after several minutes at 320°C .
- 450°C few minutes \rightarrow nothing changing
- 500°C little change: some inclusions, limited sized.
- 550°C " "

Read and Understood By

RD
Signed

Date

Peter J. Tammoh
Signed

Date

- go to $600^{\circ}\text{C} \Rightarrow$ flash crystallization $\approx 650-700^{\circ}\text{C}$ (At specimen Edge)
(NEED TO WATCH EDGES first.)

43463 D.P. 82 on 3.246

64 " "

65 " "

66 " "

67 20kX

68 100kX

69 100kX

- put in a new 13% specimen and stay near corner this time. (NB)
count 5 min intervals

- looking at specimen corner
- ramp to 200°C few minutes okay
- 400°C few minutes
- 450°C okay
- 500°C okay
- 558°C activity! definite

550°C 13% Si

- next: put in 24% Si specimen. (Sample edge)

- Amorphous

- 150°C no activity

- 200°C no activity $\rightarrow 250^{\circ}\text{C}$ no act.

- 300°C "

- 350°C "

- 400°C "

- 450°C

- 500°C

- 550°C "

- 600°C no remarkable behavior

- 650°C "

- 700°C "

- 750°C

- 800°C

- low density of nucleation (very little)
- some crystalline diffraction pattern but VERY little contrast + amorphous rings

43470 S. 088 82 on d.p. 8900

Read and Understood By

71

72

73

Peter J. Farnish

Signed

Date

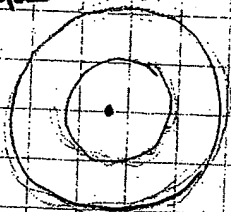
R.D.

Signed

Date

→ repeat in diffracton mode: raw sample

As dip



As by 43474 D2R 82 cm 5.211

75 5.211

500°C 5 min 76

5.211

77

} no visible DD/image change

55 °C 5 min 78

5.211

79

} "

600°C 5 min 80

5.211

81

} "

650°C 5 min 82

5.211

83

} "

700°C 5 min 84

5.211

85

} Rings appear (but weak!!)
(cannot VISUALLY see grains!!)

700°C 10 min 84

5.206

85

} "

750°C 5 min 86

87

grains easier to see, but
my alignment sucks
Sharper Rings
(VERY small, hard to see at 150KX)

Conclude:

13% $n = 2.35$ 550°C

27% $n = 2.2$ 700°C

RP
Signed

Read and Understood By

Peter J. Tancak
Signed

Date

Date

1

Document E

Pending Claims Under Consideration

1. An optical coating for a substrate, comprising:
 - a first anti-reflection layer of a dielectric;
 - a first metallic layer over the first anti-reflection layer; and
 - a second anti-reflection layer of a dielectric over the first metallic layer;wherein at least one of the first anti-reflection layer and the second anti-reflection layer comprises an amorphous material, the amorphous material comprising titanium oxide and an additive, wherein the additive in an oxidized state does not form a solid solution with the titanium oxide.
2. The optical coating according to claim 1, wherein the additive is selected from a group consisting of silicon, aluminum, bismuth, gadolinium, tantalum, zinc, and any combination thereof.
3. The optical coating according to claim 1, wherein the first metallic layer comprises silver.
4. The optical coating according to claim 1, further comprising a barrier layer between the first anti-reflection layer and the first metallic layer.
5. The optical coating according to claim 1, further comprising a barrier layer between the first metallic layer and the second anti-reflection layer.
6. The optical coating according to claim 4 or 5, wherein the barrier layer comprises a material selected from a group consisting of titanium, nickel-chromium, aluminum, and zinc.
16. A method of coating a substrate, comprising:
 - depositing a first anti-reflection layer of a dielectric over a substrate;
 - depositing a metallic layer over the first anti-reflection layer; and
 - depositing a second anti-reflection layer of a dielectric over the metallic layer;

wherein at least one of the first anti-reflection layer and the second anti-reflection layer comprises an amorphous material, the amorphous material comprising titanium oxide and an additive, wherein the additive in an oxidized state does not form a solid solution with the titanium oxide.

17. The method of claim 16, further comprising heating the coated substrate to a temperature higher than a heat-treatment temperature of the substrate after said depositing of the first anti-reflection layer, the metallic layer, and the second anti-reflection layer.

18. The method of claim 16, wherein at least one of the depositing a first anti-reflection layer, the depositing a metallic layer, and the depositing a second anti-reflection layer comprises sputtering.

19. The method of claim 16, wherein at least one of the depositing a first anti-reflection layer and the depositing a second anti-reflection layer comprises sputtering, in an oxygen environment, a target comprising titanium and the additive.

20. The method of claim 16, wherein at least one of the depositing a first anti-reflection layer and the depositing a second anti-reflection layer comprises sputtering, in an oxygen environment, a first target comprising titanium and a second target comprising the additive.

21. The method of claim 16, wherein the additive is selected from a group consisting of silicon, aluminum, bismuth, gadolinium, tantalum, zinc, and any combination thereof.